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Fabrication and Process Optimization of Wafer-scale Silicon Nanopillar SERS Substates

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Introduction

A simple approach for mass-production of wafer-scale Ag capped Si SERS nanopillars is presented and optimized. Recorded SERS spectra exhibit uniform E-field enhancement properties while retaining low background signals over large surface areas (>cm^2). 100 pM of trans-1,2-bis(4-pyridyl)-ethylene (BPE) can be detected.

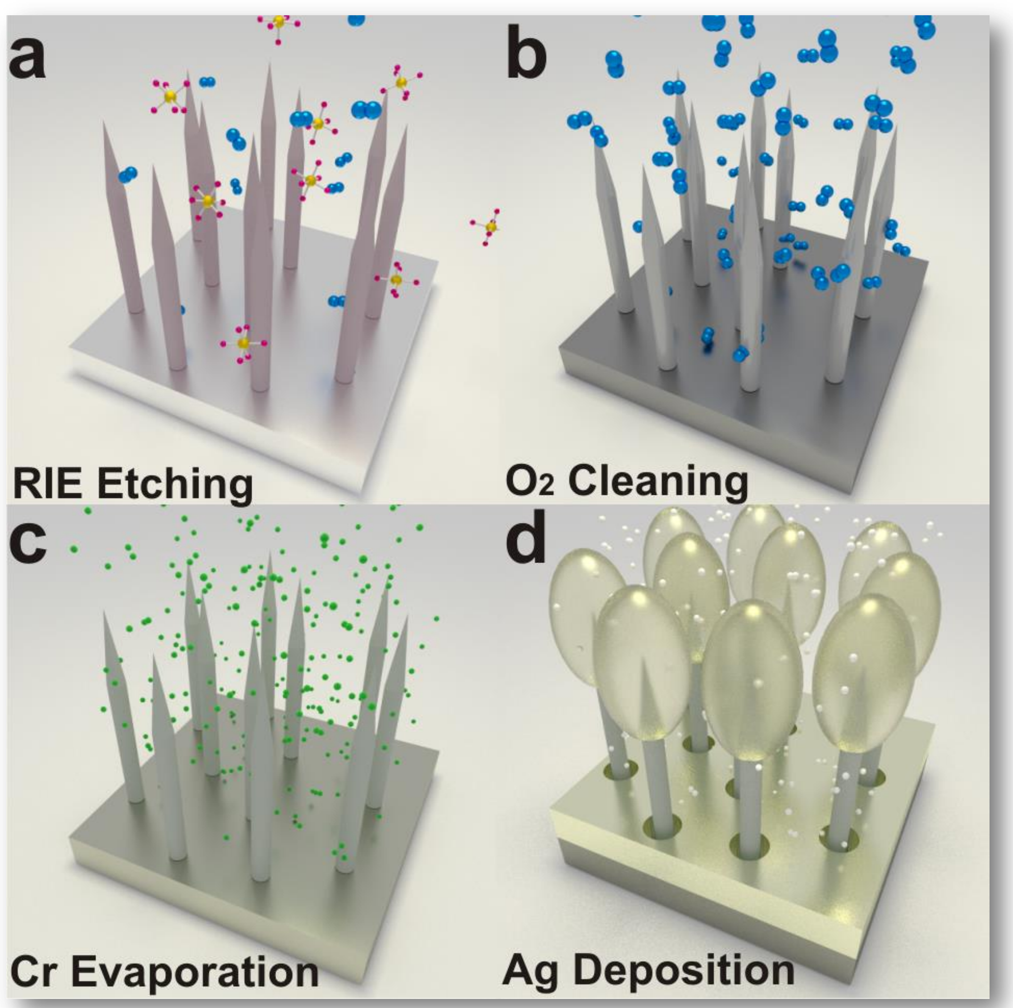
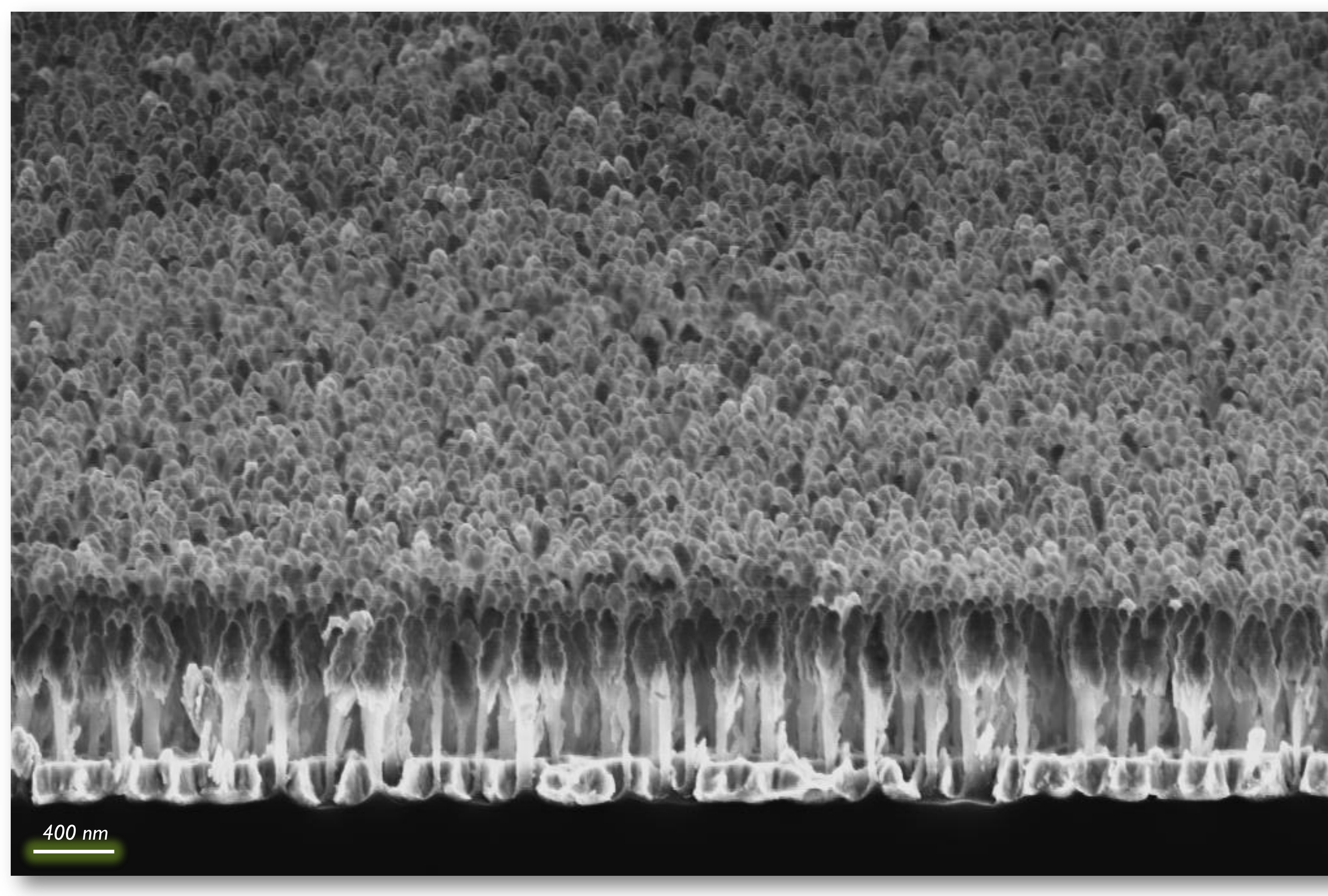


***Figure 1**. Process steps for Ag NP arrays. (a) Vertically standing Si pillars produced using maskless RIE, r ≈ 20±3 nm, h ≈ 300-1200 nm, ρNP ≈ 18±2 pillars/μm^2. (b) Si plasma etching induced surface contaminations are removed using O_2-plasma, t = 0 – 10 min. (c) Deposition of Cr adhesion layer to further reduce SERS background. (d) Evaporation of Ag, D_{Ag} = 100 – 300 nm.*



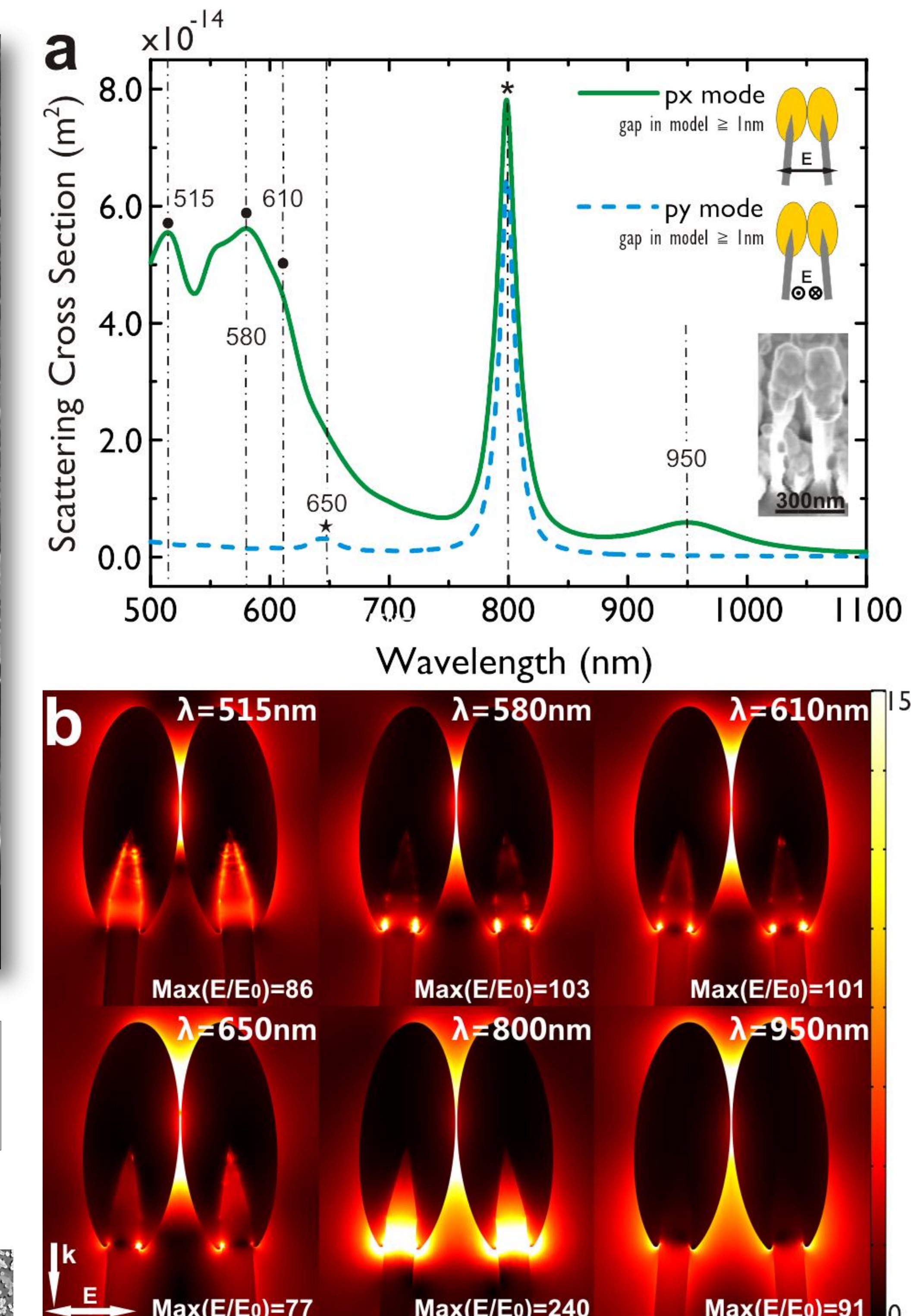


***Figure 2**. Above: SEM image of the Ag@Si nanopillar surface. Right: (a) Calculated scattering cross section of a Ag@Si nanopillar dimer under different E-field polarization directions. Inset: an SEM image of a Ag@Si pillar dimer. (b) E-field enhancement factor distributions at different excitation wavelengths.*

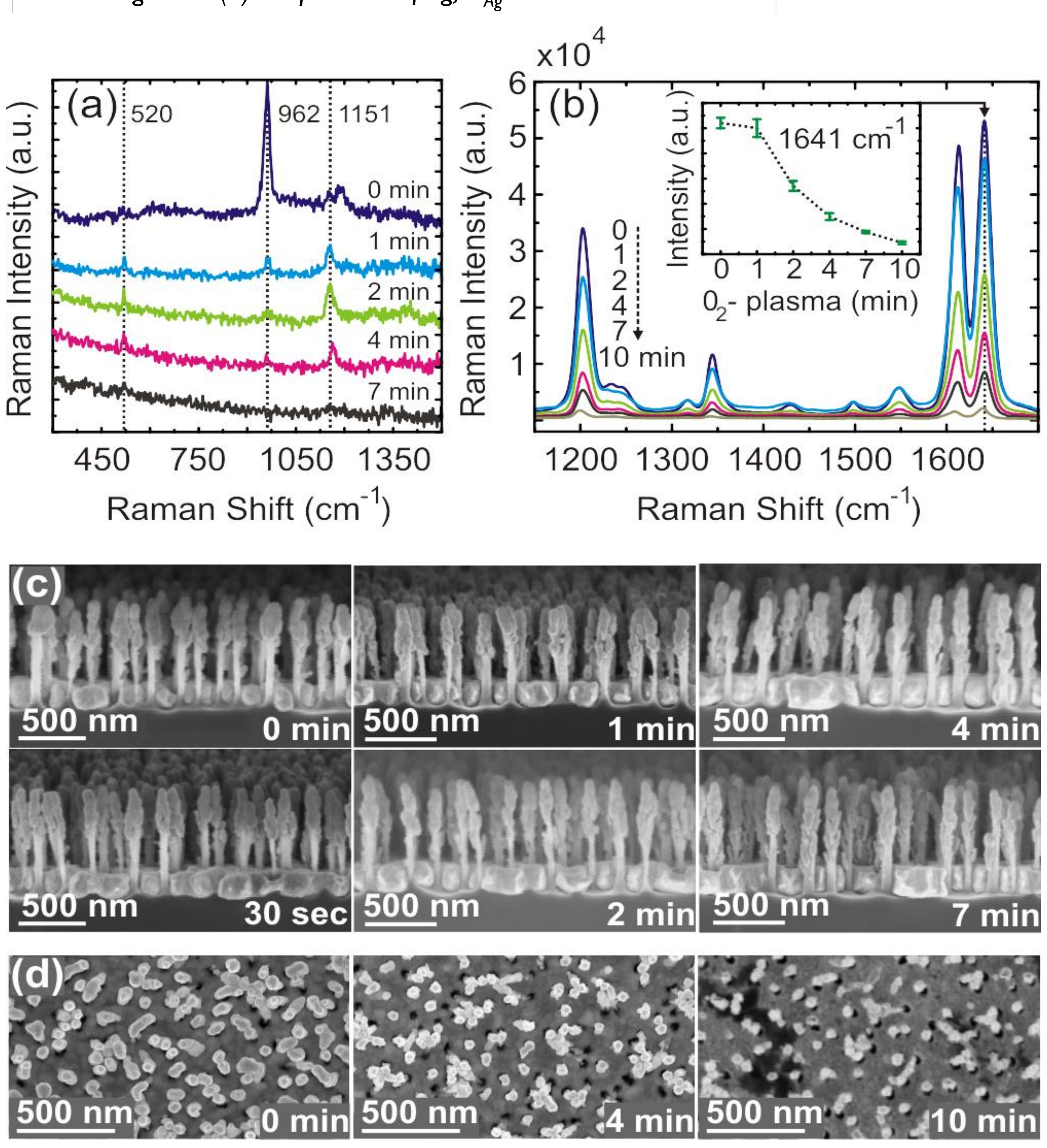


***Figure 3**. Representative SERS spectra and SEM images of the O_2-plasma treated Ag NP arrays before, (a) (c), and after, (b) (d), exposure to 1 μL of 10 mM BPE in ethanol. Solvent drying pulls Ag NPs together forming nanoclusters of varying size.*

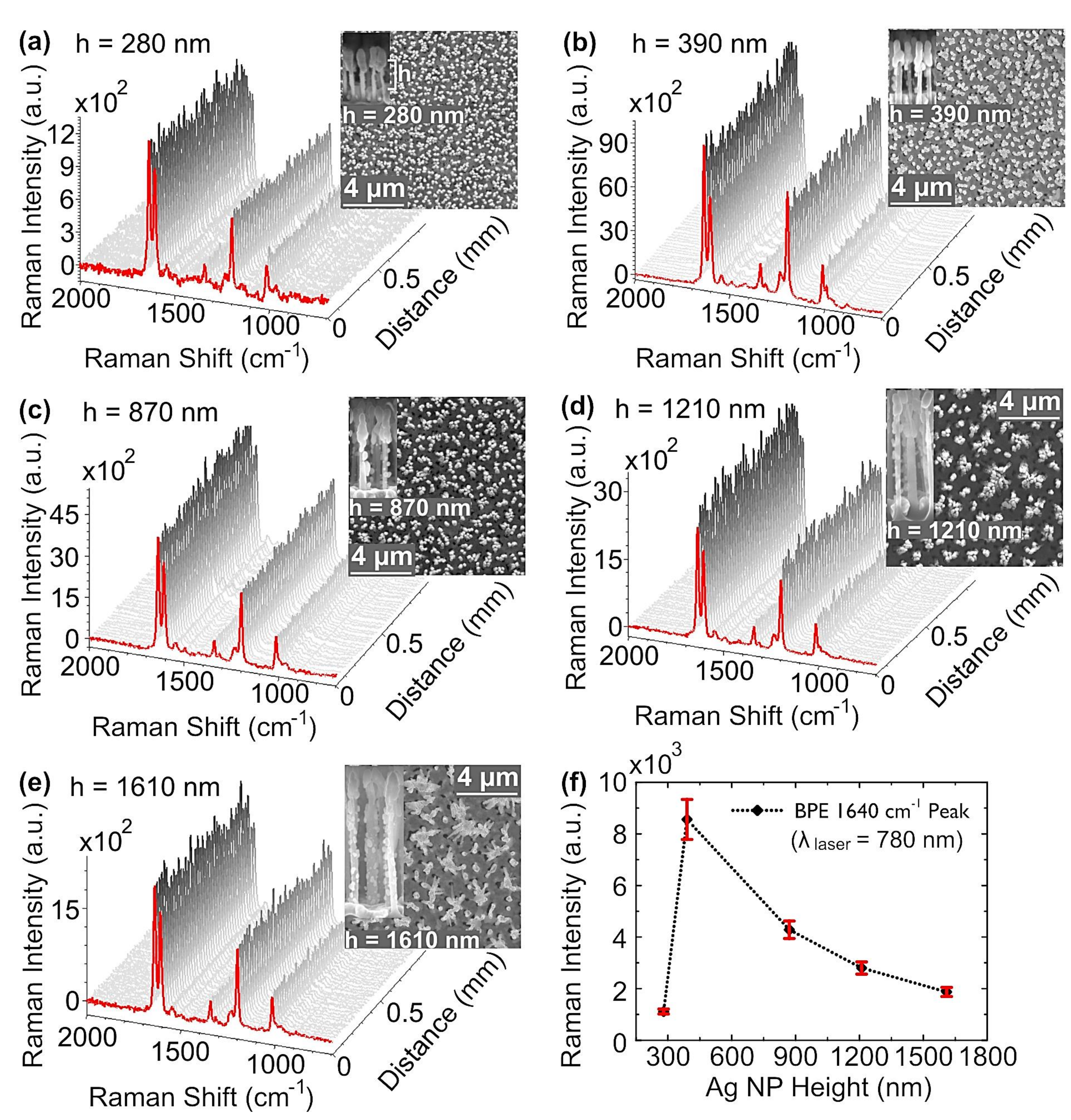


***Figure 4**. (a) - (e) Line series SERS spectra of 10 mM BPE deposited on Ag@Si NP structures with varying height (hp ≈ 280-1610 nm). SEM images illustrate the corresponding Ag NP structures (side view) and clustering of Ag NP after exposure to BPE (top view). (f) SERS intensity plots for 1641 cm^{-1} band as a function of NP height averaged by 100 spectra.*

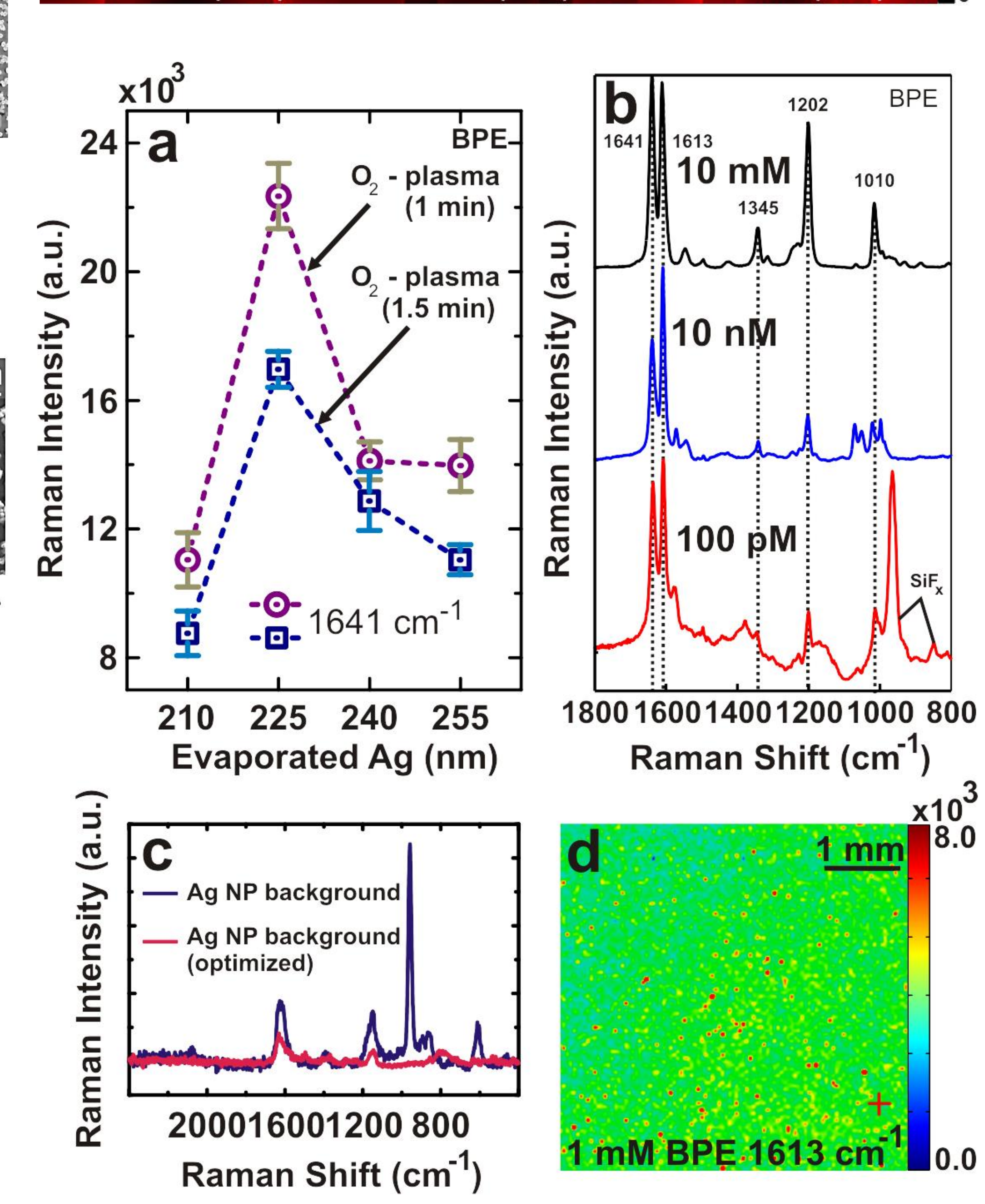


***Figure 5:** (a) Summary of SERS spectra of 10 mM BPE for substrates with varying Ag metal thickness and O_2-plasma exposure time. (b) SERS spectra of BPE recorded by optimized NPs that exhibit highest SNR. (c) A comparison between SERS background of standard and optimized Ag NP structures (after leaning). (d) Evaluation of SERS signal uniformity using the optimized substrate.*

Discussion

- FEM results in figure 2 show that the most prominent resonance mode is located in the near-infrared spectral region and contributes most to the SERS performance as well as the background of Ag NPs.
- Figure 3 shows that O_2-plasma exposure reduces the background signal. However process parameters should be carefully chosen to prevent decrease of the EF. Figure 4 shows the optimization of EF by varying the average height of the Ag NP structures.
- Figure 5 shows that a further optimized substrate by varying thickness of Ag evaporated is able to detect 100 pM BPE showing a spectrum which contains five clear Raman vibration modes. The substrate also exhibits high EF uniformity with standard deviations of ~14% across a 5 mm x 5 mm chip.

Conclusion

A simple approach for mass-production of wafer-scale Ag capped Si SERS nanopillars is presented. Optimization is done to improve SNR of the substrate. Experimental findings suggest that the Ag NP substrates are strong candidates for obtaining a reliable SERS sensing at ultra-low concentrations. The fabrication process is simple, cost-effective, CMOS compatible and could be suitable for mass-production in standard IC foundries utilizing even larger Si carrier wafers.

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Reference:

[1] M. S. Schmidt, J. Hübner, A. Boisen, Large Area Fabrication of Leaning Silicon Nanopillars for Surface Enhanced Raman Spectroscopy, Advanced Materials. 2012, 24, 11.

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